

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 10377 (1982): para-Phenylenediamine [PCD 19: Cosmetics]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



Indian Standard
SPECIFICATION FOR
para-PHENYLENEDIAMINE

UDC 547.553.1 : 665.585.32



© Copyright 1983

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR *para*-PHENYLENEDIAMINE

Cosmetics Sectional Committee, PCDC 19

<i>Chairman</i>	<i>Representing</i>
DR S. S. GOTHOSKAR	Directorate General of Health Services, New Delhi
<i>Members</i>	
SHRI N. C. CHATTERJEE	National Test House, Calcutta
SHRI V. G. DESHPANDE	Ciba-Geigy of India Ltd, Bombay
DR S. S. KARMARKAR (<i>Alternate</i>)	
SHRI W. M. FERNANDES	Food & Drug Administration, Maharashtra State, Bombay
DR S. N. IYER	Johnson & Johnson Ltd, Bombay
SHRIMATI USHA R. JOSHI (<i>Alternate</i>)	
DR G. L. MADAN	Hindustan Lever Ltd, Bombay
SHRI GURDEEP SINGH (<i>Alternate</i>)	
SHRI R. C. MEHTA	Drugs Control Administration, Government of Gujarat, Ahmadabad
SHRI J. P. GANATRA (<i>Alternate</i>)	
SHRI N. A. NIMBALKAR	Fragrance & Flavour Association, Bombay
SHRI F. F. PATANWALA	E. S. Patanwala, Bombay
SHRI N. K. SANYAL (<i>Alternate</i>)	
SHRI S. RAMASWAMY	Directorate General of Technical Development, New Delhi
SHRI S. N. AGARWAL (<i>Alternate</i>)	
SHRI K. S. RAO	Swastik Household and Industrial Products Pvt Ltd, Bombay
SHRI C. R. KRISHNAMOORTHY (<i>Alternate</i>)	
SHRI D. J. RIBEIRO	Lakmé Limited, Bombay
SHRI B. S. BARVE (<i>Alternate</i>)	
SHRI A. C. ROY	Indian Soap & Toiletries Makers' Association, Bombay
SHRI P. ROY	Bengal Chemical & Pharmaceutical Works Ltd, Calcutta
DR A. N. BASU (<i>Alternate</i>)	
DR S. K. ROY	Central Drugs Laboratory, Calcutta
DR P. C. BOSE (<i>Alternate</i>)	
SHRI V. SITARAM	Basic Chemicals, Pharmaceuticals & Cosmetics Export Promotion Council, Bombay
SHRI I. SUNDARESH (<i>Alternate</i>)	

(Continued on page 2)

© Copyright 1983

INDIAN STANDARDS INSTITUTION

This publication is protected under the *Indian Copyright Act* (XIV of 1957) and reproduction in whole or in part by any means except with written permission of the publisher shall be deemed to be an infringement of copyright under the said Act.

(Continued from page 1)

Members

SHRIMATI USHA SUKTHANKAR	Consumer's Guidance Society, Bombay
SHRIMATI USHA KAMERKAR (<i>Alternate</i>)	
SHRI M. S. THAKUR	Godrej Soaps Ltd, Bombay
SHRI N. G. IYER (<i>Alternate</i>)	
SHRI M. S. SAXENA,	Director General, ISI (<i>Ex-officio Member</i>)
Director (P&C)	

Representing

Secretary

SHRI T. R. RAJAGOPALAN
Deputy Director (P&C), ISI

Raw Materials and GRAS List Subcommittee, PCDC 19 : 1

Convener

SHRI D. J. RIBEIRO Lakmé Ltd, Bombay

Members

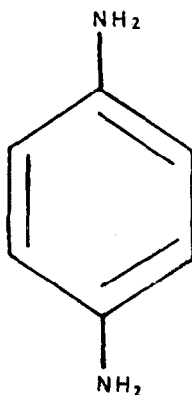
SHRI B. S. BARVE (<i>Alternate to</i> Shri D. J. Ribeiro)	
ASSISTANT COMMISSIONER (COSMETICS)	Food & Drug Administration, Maharashtra State, Bombay
SENIOR SCIENTIFIC OFFICER CLASS I (CHEM) (<i>Alternate</i>)	
SHRI V. G. DESHPANDE	Ciba-Geigy of India Ltd, Bombay
SHRI J. P. GANATRA	Drugs Control Administration, Gujarat State, Ahmadabad
SHRI R. D. DEODHAR (<i>Alternate</i>)	
DR S. N. IYER	Johnson & Johnson Ltd, Bombay
SHRIMATI USHA R. JOSHI (<i>Alternate</i>)	
DR G. L. MADAN	Hindustan Lever Ltd, Bombay
SHRI GURDEEP SINGH (<i>Alternate</i>)	
SHRI K. S. RAO	Swastik Household & Industrial Products Pvt Ltd, Bombay
SHRI C. R. KRISHNAMOORTHY (<i>Alternate</i>)	
SHRI K. L. RATHI	Sudarshan Chemical Industries Ltd, Pune
SHRI U. N. LIMAYE (<i>Alternate</i>)	
DR S. K. Roy	Central Drugs Laboratory, Calcutta
DR P. C. BOSE (<i>Alternate</i>)	
DR N. D. SHAH	Geoffrey Manners & Co Ltd, Bombay
DR M. M. DOSHI (<i>Alternate</i>)	
SHRI NAVINCHANDRA R. SHAH	Devarsons Pvt Ltd, Ahmadabad
SHRI VINODCHANDRA K. MEHTA (<i>Alternate</i>)	
SHRI M. S. THAKUR	Godrej Soaps Ltd, Bombay
SHRI N. C. IYER (<i>Alternate</i>)	

Indian Standard
SPECIFICATION FOR
para-PHENYLENEDIAMINE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 24 November 1982, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 *Para*-Phenylenediamine $C_6H_8N_2$ is an important chemical which is used in the manufacture of hair dyes. It is also known as 1,4-phenylenediamine; 1,4-benzenediamine, *p*-diaminobenzene or Ursol D. It is represented by the following structural formula:



(Molecular Mass 108.14)

para-PHENYLENEDIAMINE

0.3 The use of *para*-Phenylenediamine for hair dyes, shall be in accordance with the provisions of the Drugs and Cosmetics Act, as amended from time to time and up dated.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in

accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for *para*-phenylenediamine for cosmetic industry.

2. REQUIREMENTS

2.1 Description — The material shall be crystalline.

2.2 Colour — The colour of the material shall be as agreed to, lightgrey to black, which darkens on exposure to air.

2.3 Solubility — Sparingly soluble in cold water, soluble in alcohol, chloroform, ether and mineral acids.

2.4 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR *para*-PHENYLENEDIAMINE FOR COSMETIC INDUSTRY

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL NO. IN	
			Indian Standard	Appendix
(1)	(2)	(3)	(4)	(5)
i)	Melting range, °C	139.5 to 141.5	IS : 5762-1970*	—
ii)	Ash content, percent by mass, <i>Max</i>	0.1	—	A-1
iii)	<i>para</i> -Phenylenediamine con- tent percent by mass, <i>Min</i>	95.0	—	A-2
iv)	Iron (Fe), ppm, <i>Max</i>	100	—	A-3

*Methods for determination of melting point and melting range.

*Rules for rounding off numerical values (*revised*).

3. PACKING AND MARKING

3.1 The material shall be packed in suitable air-tight containers preferably amber coloured air-tight bottles.

NOTE — The chemical discolours in air, it is essential to keep it away from light and atmosphere in air-tight containers.

3.2 Marking — The containers shall be marked with the following:

- a) Name of the material;
- b) Tare, gross and net mass of the material;
- c) Name of the manufacturer and his recognized trade-mark, if any
- d) Batch No. in code or otherwise, to enable the date of manufacture to be traced from records; and
- e) Any other information required by statutory authorities.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

CAUTION — *para*-Phenylenediamine may cause skin irritation in certain cases and so the following preliminary test should first be made:

‘Cleanse a small area of skin behind the ear or upon the inner surface of the forearm, using either soap and water or alcohol. Apply a small quantity of the hair dye as prepared for use to the area and allow it to dry. After 24 hours, wash the area gently with soap and water. If no irritation or inflammation is apparent, it may be assumed that no hypersensitivity to the dye exists. The test should, however, be carried out before each and every application.’

The material should not be used for products used for dyeing the eyelashes or eyebrows as such a use may cause blindness.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in IS : 3958-1966*.

4.2 Tests for all the requirements shall be carried out on a composite sample.

4.3 The method for preparing representative test samples of the material and the criteria for conformity shall be as prescribed in 6 of IS : 3958-1966*. The lot shall be declared as conforming to the requirements of this specification if all the test results on the composite sample meet the relevant specifications requirements.

APPENDIX A

[Table 1, Items (ii), (iii) and (iv)]

A-1. DETERMINATION OF ASH CONTENT

A-1.0 Outline of the Method — A known quantity of the material is ignited at 600°C and the ash cooled and weighed to constant mass.

A-1.1 Apparatus

A-1.1.1 Platinum or Silica Crucible — provided with a lid.

A-1.1.2 Furnace — preferably with automatic temperature control device.

A-1.1.3 Desiccator

A-1.2 Procedure — Incinerate 2 to 3 g of the sample in a tared platinum or silica crucible and then ignite at dull red heat (approximately at 600°C). Cool in a desiccator and weigh till constant mass is obtained.

A-1.3 Calculation

$$\text{Ash, percent by mass} = \frac{m \times 100}{M}$$

where

m = mass in g of the residue obtained, and

M = mass in g of the sample taken for the test.

*Methods of sampling cosmetics and toilet goods.

A-2. DETERMINATION OF *para*-PHENYLENEDIAMINE CONTENT

A-2.1 Outline of the Method — This method estimates the *para*-phenylenediamine as diacetyl derivative of *para*-phenylenediamine.

A-2.2 Apparatus — G₄ sintered glass crucibles.

A-2.3 Reagents — 1. Chloroform, and
2. Acetic anhydride.

A-2.4 Procedure — Weigh accurately 0.2 to 0.3 g of the sample into a 100 ml beaker. Add about 30 ml of chloroform. Heat on a water bath while stirring. Filter through a G₄ sintered glass crucible and collect the filtrate in a flask. Care should be taken to retain the solids in the beaker. Repeat extraction with chloroform at least for three more times taking 25 ml each time or until the filtrate is colourless (maximum six extractions).

A-2.5 Remove the flask and transfer the filtrate to a 250-ml beaker. Rinse with few small portions of chloroform. Evaporate chloroform to about 25 ml and add 1 ml of acetic anhydride slowly, with stirring. Let it stand for one hour and filter on a weighed G₄ sintered glass crucible. Wash beaker and precipitate with three or more, 5 ml portions of chloroform. Carefully remove last traces of precipitate from the beaker. Dry the crucible to constant mass at 120°C and weigh the precipitate.

A-2.6 Calculation

$$p\text{-Phenylenediamine, percent by mass} = \frac{M_2 \times 0.5626 \times 100}{M_1}$$

where

M_2 = mass in g of the precipitate, and

M_1 = mass in g of the sample taken.

A-3. DETERMINATION OF IRON CONTENT

A-3.1 Apparatus

A-3.1.1 Nessler Cylinders — 50 ml capacity.

A-3.2 Reagents

A-3.2.1 Ammonia Solution Iron Free — Dilute 37.5 ml of strong ammonia solution to 100 ml with distilled water. This solution contains

approximately 10 percent ammonia (m/m). The solution should comply with the following additional test:

Evaporate 5 ml nearly to dryness on a water bath. Add 40 ml of water, 2 ml of a 20 percent m/v solution of citric acid and 2 drops of thioglycollic acid. Mix, make alkaline with ammonia solution, and dilute to 50 ml with water; no pink colour is produced.

A-3.2.2 Citric Acid Solution 20 percent (m/v) Solution of Iron Free Citric Acid in Water — The solution should comply with the following additional test:

Dissolve 2 g of the solution in 40 ml of water, add 2 drops of thioglycollic acid and mix. Make alkaline with ammonia solution and dilute to 50 ml with water; no pink colour is produced.

A-3.2.3 Concentrated Hydrochloric Acid — See IS : 265-1976*. Hydrochloric acid should comply with the following additional test:

Evaporate 5 ml on a water bath nearly to dryness. Add 40 ml of water, 2 ml of a 20 percent m/v solution of citric acid and two drops of thioglycollic acid, and mix. Make to 50 ml with water; no pink colour is produced.

A-3.2.4 Sodium Hydroxide Solution 4 percent (m/v) — Sodium hydroxide solution should comply with the following additional test:

To 5 ml, add 2 ml of 20 percent m/v solution of citric acid and 2 drops of thioglycollic acid and mix. Make alkaline with ammonia solution and dilute to 50 ml with water; no pink colour is produced.

A-3.2.5 Thioglycollic Acid — iron-free.

A-3.2.6 Standard Iron Solution — Dissolve 0.173 g of ferric ammonium sulphate in 100 ml of water, add 5 ml of dilute hydrochloric acid and dilute to 1 000 ml. One millilitre of this solution contains 0.02 mg of iron.

A-3.3 Procedure

A-3.3.1 Take 10 g of the sample in a silica evaporating dish. Wet with 5 ml of sodium hydroxide solution and evaporate to dryness. Ash the sample in a muffle maintained at 600-700°C. After the sample is coded down in a desiccator, add 5 ml of concentrated hydrochloric acid and swirl until all of the ash is netted with the acid. Cover the dish, heat to boiling, dilute with 10 ml distilled water and reheat to boiling. Cool and transfer to a 50-ml volumetric flask. Make to volume with the distilled water.

*Specification for hydrochloric acid (second revision).

A-3.3.2 Prepare standards from the standard iron solution by the following method:

Take 1 ml, 1.2 ml, 1.4 ml, 1.6 ml, 1.8 ml and 2 ml of standard iron solution (**A-3.2.6**) in nessler tubes. Add 2 ml of 20 percent *m/v* solution of citric acid, 2 drops of thioglycollic acid and mix. Make alkaline with ammonia solution and dilute to 50 ml with water. Allow to stand for five minutes. Pipette a certain volume of the solution from **A-3.3.1** in place of standard iron solution in the nessler tube and proceed exactly as given in **A-3.3.2**. Compare the colour produced with the standards prepared above. If the colour is darker than the one made with 2 ml standard iron solution, take another volume of solution from **A-3.3.1** so that the colour produced is within the range of standards.

A-3.3.3 Calculations

$$\text{Iron (as Fe), ppm} = \frac{A \times 100}{B}$$

where

A = volume of standard solution in ml to match *B*, and

B = volume of solution from **A-3.3.1** matching with *A*.

INDIAN STANDARDS
ON
COSMETIC RAW MATERIALS

IS:

- 263-1977 Boric acid (*third revision*)
- 918-1968 Calcium carbonate, precipitated, for cosmetic industry (*first revision*)
- 1462-1977 Talc for cosmetic industry (*second revision*)
- 1463-1977 Kaolin for cosmetic industry (*second revision*)
- 1767-1980 Dicalcium phosphate for dentifrice (*second revision*)
- 2519-1977 Calcium stearate for cosmetic industry (*first revision*)
- 2520-1977 Zinc stearate for cosmetic industry (*first revision*)
- 2521-1977 Magnesium stearate for cosmetic industry (*first revision*)
- 2528-1977 Magnesium carbonate for cosmetic industry (*first revision*)
- 2529-1977 Magnesium oxide for cosmetic industry (*first revision*)
- 2850-1977 Zinc oxide for cosmetic industry (*first revision*)
- 2851-1978 Titanium dioxide for cosmetic industry (*first revision*)
- 3986-1973 Sodium lauryl sulphate for cosmetic industry (*first revision*)
- 3987-1977 Sorbitol solution (70 percent) (*first revision*)
- 4028-1977 Beeswax, bleached, for cosmetic industry (*first revision*)
- 4236-1977 Glyceryl monostearate for cosmetic industry (*first revision*)
- 4652-1980 Ethyl *p*-hydroxybenzoate for cosmetic industry (*first revision*)
- 4653-1977 Methyl *p*-hydroxybenzoate for cosmetic industry (*first revision*)
- 4887-1980 Petroleum jelly for cosmetic industry (*first revision*)
- 5340-1981 Lanoline, anhydrous, for cosmetic industry (*first revision*)
- 5356-1977 *iso*-propyl myristate for cosmetic industry (*first revision*)
- 6333-1977 Propyl *p*-hydroxybenzoate for cosmetic industry (*first revision*)
- 6334-1980 Butyl *p*-hydroxybenzoate for cosmetic industry (*first revision*)
- 7101-1973 Coconut diethanolamide
- 7299-1974 Mineral oil for cosmetic industry
- 9601-1980 Sodium silicate for cosmetic industry
- 9681-1980 Stearic acid for cosmetic industry
- 9830-1981 Water soluble sodium carboxy methyl cellulose for cosmetic industry
- 9831-1981 Sodium hydroxide for cosmetic industry